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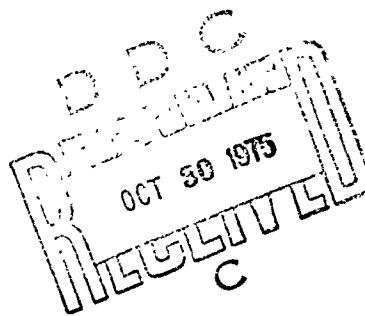
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DETERMINATION OF BARIUM AND STRONTIUM PEROXIDES
(ACTIVE OXYGEN) IN IGNITERS IN SMALL
ARMS TRACER AMMUNITION

August 1975



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Technical Support Directorate

U.S. ARMY ARMAMENT COMMAND
FRANKFORD ARSENAL
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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER FA-TT-75059	2. GVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) DETERMINATION OF BARIUM AND STRONTIUM PEROXIDES (ACTIVE OXYGEN) IN IGNITERS IN SMALL ARMS TRACER AMMUNITION.		5. TYPE OF REPORT & PERIOD COVERED 91 Technical Test Report
6. PERFORMING ORG. REPORT NUMBER		7. CONTRACT OR GRANT NUMBER(s)
8. PERFORMING ORGANIZATION NAME AND ADDRESS Commander FRANKFORD ARSENAL, ATTN: SARFA-TSE-M-64-1 Philadelphia, PA 19137		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS AMS Code: 53970M6350 PRON: A1-5-K005-F6-NH
11. CONTROLLING OFFICE NAME AND ADDRESS Commander Army Materials and Mechanics Research Center ATTN: AMXMR-MO, Watertown, MA 02172		12. REPORT DATE Aug 1975
13. NUMBER OF PAGES 20		14. SECURITY CLASS. (of this report) UNCLASSIFIED
15. DECLASSIFICATION/DOWNGRADING SCHEDULE N/A		16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Barium peroxide Igniters Strontium peroxide Small Arms Oxygen Tracers Active oxygen Ammunition		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A method is proposed for the determination of barium and strontium peroxides (active oxygen) in igniters in small arms ammunition. The sample is treated with dilute hydrochloric acid (1 to 9) which dissolves the magnesium powder almost instantaneously and then dissolves the barium or strontium peroxides within 10 to 30 minutes. The solution is then filtered to remove the organic substances (calcium resinate, Parlon, toluidine red toner, and zinc stearate) and the peroxide is determined by the (continued)		

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20. ABSTRACT: (continued)

titanium peroxide color. It appears that igniter mixes and igniters used in small arms tracer ammunition deteriorate with storage.

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INTRODUCTION

Tracer ammunition for both small arms and artillery are used for determining range and directing fire. The tracer compositions in such ammunition are set off by igniters which may contain barium or strontium peroxides (oxidizers), calcium resinate, Parlon, or zinc stearate (binders and fuels), magnesium powder (fuel), colloidine red toner (identifying coloring agent and fuel). It has been noted by ammunition experts for many years that the igniters (and consequently the rounds of tracer ammunition) are inclined to lose their effectiveness with time and it has been theorized that this deterioration is caused by decomposition of the peroxide. However, this has never been proved because of the lack of satisfactory analytical methods.

In view of the need, this arsenal undertook an investigation to develop a method for the analysis of peroxide (active oxygen) in igniters in small arms ammunition. Many methods that had been previously used for determining peroxide in various materials were considered. The most common method for determining peroxide is the titrimetric permanganate method.^{1-5*} Other titrimetric methods that have been used involve ceric sulfate, potassium iodide, sodium thiosulfate, titanous chloride, and sodium nitrite.^{1, 5, 6*} Another method for peroxide is the eudiometric method whereby the volume of oxygen generated on decomposition is measured.^{6*} Spectrophotometric methods that have been proposed include the use of the titanium peroxide,^{5-9*} permanganate decolorization,^{5, 6*} ferric-thiocyanate,^{6*} starch-iodide,^{6, 10*} and ferrous-orthophenanthroline^{11*} methods.

The potassium permanganate titrimetric method was found to be unreliable for the determination of peroxides in igniters because of interference from organic matter and magnesium powder. It is possible that some other titrimetric reagent could be used; however, this approach was not pursued because a titrimetric method would not be applicable in any case to the accurate determination of the small amount of igniter to be found in ammunition. The eudiometer method was not applicable because of interference from hydrogen generated by the magnesium. It seemed, therefore, that a spectrophotometric method would be the most feasible means for determining peroxide in igniters. The titanium peroxide spectrophotometric method was selected as the most useful.

* See References, page 11.

EXPERIMENTAL

Apparatus and Reagents

Beckman Model DU spectrophotometer (or equivalent), 1-cm cell.

Titanium solution. Weigh 2.00 g of titanium metal (sponge type) into a 250-ml beaker, add 90 ml of hydrochloric acid, cover with a watch glass, and warm on the hot plate to dissolve. Maintain the volume of hydrochloric acid at 75-90 ml during the dissolution process. After the metal has completely dissolved, add 1 ml of nitric acid and boil for 1 minute. Cool and dilute to 200 ml in a volumetric flask.

Barium peroxide, MIL-B-153A, Grade B²* (the sample used in this work contained 8.9% active oxygen).

Strontium peroxide, MIL-S-512B, Grade B³* (the sample used in this work contained 12.7% active oxygen).

Magnesium powder, MIL-M-382E, Type III, Granulation 12, nominal mesh size 120-200.¹²*

Calcium resinate, MIL-C-20470A, Type II.¹³*

Parlon (chlorinated rubber), MIL-R-60671.¹⁴*

Zinc stearate, USP.¹⁵*

Toluidine red toner, TT-P-445.¹⁶*

Preparation of Calibration Curves

Barium peroxide. Transfer 0.20-0.21 g of barium peroxide (weighed to 0.1 mg) to a 150-ml beaker. Add 100 ml of dilute hydrochloric acid (1 to 9) and allow to stand with occasional stirring until the sample has dissolved (10 to 30 minutes). Dilute to 250 ml in a volumetric flask. Transfer 5.0, 10.0, 15.0, 20.0, and 25.0 ml aliquots to 100-ml volumetric flasks and dilute to about 35 ml with water. Add sufficient hydrochloric acid to bring the hydrochloric acid content in each flask to 5 ml. Dilute to about 75 ml with water and add 5.0 ml of titanium solution. Dilute to the mark and within 1 hour, measure the absorbance at 410 nm against the reagent blank. Plot mg of barium peroxide (per 100 ml) against absorbance.

* See References, page 11.

Strontium peroxide. Transfer 0.15 - 0.16 g of strontium peroxide (weighed to 0.1 mg) to a 150-ml beaker and proceed as described for the calibration curve for barium peroxide but use 5.0, 10.0, 15.0, 17.5, and 20.0 ml aliquots. Plot mg of strontium peroxide (per 100 ml) against absorbance.

Procedure

Igniter Mixes. Transfer about 0.20 g of sample (weighed to 0.1 mg) to a 150-ml beaker. Add 100 ml of dilute hydrochloric acid (1 to 9) at one stroke. Allow to stand with occasional stirring until the barium or strontium peroxide has dissolved (10 to 30 minutes). Within 30 minutes after dissolution, filter through a Whatman No. 41 filter paper into a 250-ml volumetric flask and wash with water. Discard the filter paper and dilute to the mark in the volumetric flask. Transfer an aliquot preferably containing 13 to 20 mg of barium peroxide or 9 to 15 mg of strontium peroxide to a 100-ml volumetric flask and dilute to about 35 ml. Add sufficient hydrochloric acid to bring the hydrochloric acid content to 5 ml and dilute to about 75 ml. Add 5.0 ml of titanium solution, dilute to the mark, and within 1 hour measure the absorbance at 410 nm against the reagent blank. Determine the mg of barium or strontium peroxide by referring to the calibration curve and calculate the percent barium or strontium peroxide as follows:

$$\% \text{ BaO}_2 \text{ or } \text{SrO}_2 = \frac{\text{mg of BaO}_2 \text{ or } \text{SrO}_2 \text{ as read from curve}}{\text{g of sample in aliquot}} \times 1.01 \times 10$$

Igniters Contained in Small Arms Tracer Ammunition. Remove the bullet from the round. While working behind safety shield, cut through the exterior of the bullet jacket lengthwise on both sides with a hand-saw to just sufficient depth to reach the tracer and igniter. Exercise care during this cutting operation so as not to overheat the bullet since overheating might cause some decomposition of the peroxide and constitute a hazard. Pry off one side of the jacket with a suitable instrument. Remove the igniter mix and store it in a closed bottle. Transfer a sample to a 150-ml beaker, add 35 to 100 ml of dilute hydrochloric acid (1 to 9), allow to stand until the barium or strontium peroxide has dissolved, filter, wash, and dilute to volume in a volumetric flask (the amount of hydrochloric acid and the volume of dilution will depend on the amount of sample). Transfer an appropriate aliquot to a 100-ml volumetric flask and proceed as described for barium and strontium peroxides in igniter mixes.

DISCUSSION AND RESULTS

The magnesium metal dissolves almost instantaneously on adding 100 ml of 1 to 9 hydrochloric acid at room temperature at one stroke; then, the barium or strontium peroxide dissolves slowly in 10 to 30 minutes. The extremely rapid dissolution of the magnesium minimizes the interference of magnesium. If a more concentrated hydrochloric acid solution were used, the barium and strontium peroxides would dissolve simultaneously with the magnesium, causing lower results. The dissolution process with the 1 to 9 hydrochloric acid must be conducted at room temperature. If the solutions were cooled, the magnesium would not dissolve instantly, causing lower results. The organic matter contained in the igniters does not dissolve in the 1 to 9 hydrochloric acid and is filtered off and does not seem to interfere. In spite of all precautions, however, preliminary work with synthetic samples showed that the recoveries for peroxides were slightly low; therefore, it was decided on the basis of this preliminary work to multiply calculated result by the empirical factor 1.01.

Hydrochloric acid rather than sulfuric acid is used because the latter acid would cause precipitation of insoluble barium and strontium sulfates.

The acidity of the solution in which the titanium peroxide color is developed is about 1.8N. This acidity is not critical since the permissible acidity for titanium peroxide color is about 1.5 to 3.5N.^{17*}

Full color development was achieved with 5 ml of the titanium solution. The titanium solution used by the authors was prepared from titanium metal since that material could be readily obtained in high purity (previous investigators have prepared the solution from titanium dioxide, potassium titanium oxalate, potassium fluotitanate, and titanium sulfate).^{6-9, 17*} Nitric acid must be used in oxidizing Ti(III) to Ti(IV); oxidizing with hydrogen peroxide and subsequent boiling to destroy the peroxide caused hydrolysis of the titanium.

The calibration curves follow Beer's law (0 to about 0.75 absorbance).

All the calculations for the barium and strontium peroxides in igniters were relative to the original barium and strontium peroxides which were arbitrarily taken as 100% pure. This mode of calculation was chosen because the prime aim of the investigation was to determine whether there had been degradation of the barium or strontium peroxide in the igniters.

* See References, page 11.

If it were desired to make the calculation for the igniters in terms of active oxygen it would be first necessary to determine the active oxygen content of the barium and strontium peroxides used for preparing the calibration curves and then plot mg of active oxygen (per 100 ml) against absorbance. This would be a relatively simple matter.

The active oxygen of barium or strontium peroxide can be defined as the percent oxygen (by weight) released when the peroxide reverts to the simple oxide (eg., $2\text{BaO}_2 \rightarrow 2\text{BaO} + \text{O}_2$). Active oxygen can also be calculated as the volume of oxygen released but this is ordinarily done only with hydrogen peroxide (which is frequently characterized by the ml of oxygen at 0°C and 760 mm pressure that is liberated from 1 ml or 100 ml of solution measured at 20°C when the peroxide is completely decomposed). The theoretical active oxygen content of barium and strontium peroxides is 9.45% and 13.38%, respectively.

The military specifications for barium and strontium peroxides^{2,3} call for a minimum active oxygen content of 8.5% and 12.3%, respectively. However, according to the experiences of this arsenal over many years, the active oxygen content of the barium peroxide used in igniters varies from 8.8 - 9.0% for barium peroxide and 12.5 - 12.8% for strontium peroxide (and it does not change significantly for 2 or 3 years if the two materials are stored in air-tight containers). The cited ranges are equivalent to 93.1 - 95.2% barium peroxide and 93.4 - 95.7% strontium peroxide, respectively. Rosin⁴ and Fisher Scientific Co.¹³ give a minimum barium peroxide content of 88% and 85%, respectively, for reagent barium peroxide. There is no specification for reagent strontium peroxide.

According to experiences of this laboratory, the permanganate method for active oxygen described in the military specifications for barium and strontium peroxides^{2,3} is far superior to other titrimetric methods for active oxygen. These specification methods involve the addition of 3/4 of the amount of 0.1N potassium permanganate needed for the titration to 70 ml of acid reagent at room temperature (the acid reagent is made by dissolving 0.1 g $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$, 100 ml of perchloric acid, and 30 ml of phosphoric acid in water and diluting to 1 liter). Then, 0.35-0.40 g of the barium or strontium peroxide is added while stirring with a bar magnet stirrer and the solution is titrated again with the permanganate to a pink color that persists for 1 minute.

$$\% \text{ active oxygen} = \frac{0.800 \times \text{volume of KMnO}_4 \times \text{N of KMnO}_4}{\text{g of sample}}$$

²Military Specification MIL-B-153A, Barium Peroxide, 30 September 1964.

³Military Specification MIL-S-612B, Strontium Peroxide, 15 May 1975.

⁴J. Rosin, "Reagent Chemicals and Standards," 4th Ed., p. 68, New York, Van Nostrand, 1961.

¹³Military Specification MIL-C-20470A, Calcium Resinate, 30 September 1964.

The nominal compositions of the common igniter mixes used in small arms tracer ammunition by the US Army are shown in Table I. The results obtained for barium and strontium peroxides in synthetic igniter mixes prepared in this laboratory are shown in Table II. The results obtained for three actual production igniter mixes stored in closed containers for about a year are shown in Table III. The results obtained for an igniter in a lot of ammunition several years old are shown in Table IV.

The recoveries obtained for the synthetic samples were satisfactory (the average recovery of all the runs was 100.2%). The precision of the results for the igniter mixes and the igniter was satisfactory. The average standard deviation for the three igniter mixes was 0.70%, while the average standard deviation for the igniter in the ammunition was 1.54%. It would be expected, as indicated, that it would be more difficult to obtain a uniform sample from the igniters in the ammunition.

It is seen that two of the three actual igniter mixes showed some deterioration and the igniter in the ammunition showed marked deterioration. The data would seem to indicate that I-276 is a superior mix. The data does not show whether the dry mix process is superior to the wet mix process. In the dry mix process (used for Igniters I-276 and I-508) the ingredients are mixed dry, while in the wet mix process (used for Igniter I-280) the ingredients are mixed while dampened with carbon tetrachloride, which is then volatilized by heating at 140° F.¹⁹ The wet mix process is used because it reduces the hazard caused by the ready ignitability of the dust particles of the igniter mix. The data is insufficient to show whether barium or strontium peroxide is more stable in igniters.

SUMMARY

A method is proposed for the determination of barium and strontium peroxides (active oxygen) in igniters in small arms tracer ammunition. The sample is treated with dilute hydrochloric acid (1 to 9) which dissolves the magnesium powder almost instantaneously and then dissolves the barium or strontium peroxides within 10 to 30 minutes. The solution is then filtered to remove the organic substances (calcium resinate, Parlon, toluidine red toner, and zinc stearate) and the peroxide is determined by the titanium peroxide color. It appears that igniters and igniter mixtures used in small arms tracer ammunition deteriorate with storage.

¹⁹Ordnance Corps, Department of the Army, Frankford Arsenal, Drawings for Igniter Compositions I-136, I-280, and I-508.

RECOMMENDATIONS

It is recommended that the deterioration of igniters be investigated in greater detail to establish the following: (1) relative susceptibility of the different igniter compositions to deterioration, (2) mechanism of the decomposition, (3) effect of different storage condition on the deterioration.

Table I.
Nominal Composition of Igniters Used
in Small Arms Tracer Ammunition

<u>Igniter</u>	<u>Nominal Composition (%)</u>
I-280	76.5 SrO ₂ , 15.0 Mg, 8.5 Ca resinate
I-276	83.5 BaO ₂ , 15.0 Mg, 1.0 Zn stearate, 0.5 toluidine red toner
I-508	79.0 BaO ₂ , 14.1 Mg, 5.5 Parlon, 0.9 Zn stearate, 0.5 toluidine red toner

Table II.
Results for Barium and Strontium Peroxides in Synthetic Igniter Mixes

	<u>Present</u>	<u>Found (g)</u>	<u>Recovery (%)</u>
0.1514 g	BaO ₂ + 25 mg Mg	0.1523	BaO ₂ 100.6
0.1518 "	"	0.1523	BaO ₂ 100.3
0.1519 "	"	0.1534	BaO ₂ 101.0
0.1479 "	"	0.1474	BaO ₂ 99.7
0.1484 "	"	0.1482	BaO ₂ 99.9
0.1523 g	SrO ₂ + 25 mg Mg + 15 mg Ca resinate ^a	0.1507	SrO ₂ 99.0
0.1585 "	"	0.1555	SrO ₂ 98.1
0.1590 "	"	0.1602	SrO ₂ 100.8
0.1624 "	"	0.1613	SrO ₂ 99.3
0.1509 "	"	0.1518	SrO ₂ 100.6
0.1642 g	BaO ₂ + 30 mg Mg + 5 mg Zn stearate + 5 mg tol. red ^b	0.1632	BaO ₂ 99.4
0.1683 "	"	0.1679	BaO ₂ 99.8
0.1684 "	"	0.1679	BaO ₂ 99.7
0.1663 "	"	0.1682	BaO ₂ 101.1
0.1514 "	"	0.1548	BaO ₂ 102.2
0.1545 g	BaO ₂ + 30 mg Mg + 15 mg Parlon + 5 mg Zn stearate + 5 mg tol. red ^c	0.1553	BaO ₂ 100.5
0.1583 "	"	0.1590	BaO ₂ 100.4
0.1587 "	"	0.1589	BaO ₂ 100.1
0.1566 "	"	0.1581	BaO ₂ 101.0
0.1567 "	"	0.1578	BaO ₂ 100.7

^a Approximately I-280 composition.

^b Approximately I-276 composition.

^c Approximately I-508 composition.

Table III.
Results for Strontium and Barium Peroxides
in Actual Igniter Mixes

<u>Igniter</u>	<u>Nominal (%)</u>	<u>Results (%)</u>
I-280	76.5 SrO ₂	67.5
		66.7
		67.5
		67.4
		67.0
		Avg. 67.2
		Std. Dev. 0.36
I-276	83.5 BaO ₂	88.4
		88.4
		90.0
		88.7
		90.0
		88.8
		Avg. 89.1
		Std. Dev. 0.75
I-508	79.0 BaO ₂	72.7
		71.2
		72.7
		72.9
		73.3
		73.3
		Avg. 72.7
		Std. Dev. 1.00

Table IV.
Results for Barium Peroxide in
Igniter I-508 in Ammunition

<u>Nominal BaO₂ (%)</u>	<u>BaO₂ Found (%)</u>
79.0	57.0
	49.0
	53.8
	Avg. 53.3
	Std. Dev. 1.54

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FRANKFORD ARSENAL
Date Printed: 10 October 1975